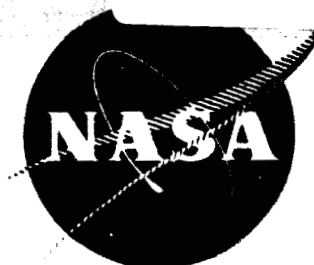


NASA CR-54152
TRW ER-6116

FACILITY FORM 602
N64-30033
(ACCESSION NUMBER)
37
(PAGES)
Cr. 54152
(NASA CR OR TMX OR AD NUMBER)

(THRU)
1
(CODE)
27
(CATEGORY)



POROUS MATRIX PHASE SEPARATOR EVALUATION

OTS PRICE

3.60 ph

\$

XEROX

MICROFILM

\$

By
KEITH M. MONTGOMERY

Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NAS 3-2518

TRW

THOMPSON RAMO WOOLDRIDGE INC.
CLEVELAND, OHIO

NASA CR-54152

TRW ER-6116

FINAL REPORT

POROUS MATRIX
PHASE SEPARATOR EVALUATION

by

Keith M. Montgomery

Prepared for

National Aeronautics and Space Administration

September 18, 1964

Contract NAS3-2518

Technical Management
NASA Lewis Research Center
Cleveland, Ohio

Spacecraft Technology
Procurement Section

R. H. Hieber
and
J. T. Kotnik

TRW ELECTROMECHANICAL DIVISION
Thompson Ramo Wooldridge Inc.
New Product Research Department
23555 Euclid Avenue
Cleveland, Ohio 44117

Foreword

This report was prepared by the New Product Research Department of the Electro-mechanical Division of Thompson Ramo Wooldridge Inc., Cleveland, Ohio on Contract No. NAS3-2518. The contract was administered by the National Aeronautics and Space Administration, with Messrs. R. H. Hieber and J. T. Kotnik acting as contract monitors.

Studies presented in this report began in March 1963, and were concluded in July 1964. William C. Davis, Senior Engineering Specialist, and Patrick J. Lawlor, Project Engineer, were responsible for the development activity carried on at TRW Inc. The chief contributors to the program were K. M. Montgomery, system analyses, design, and test and E. L. Kubiak, test support.

Porous Matrix Phase Separator
Evaluation

by
K. M. Montgomery

Abstract

30033

A porous matrix phase separator, of the type proposed for use in a cesium feed system of an ion propulsion rocket, was evaluated for plugging and deterioration in a vaporization experiment. The experiment was concluded after 2100 hours of continuous cesium boiling at 800°F. Radiographs taken periodically during the course of the experiment, and post-experiment metallographic and spectrographic examination of the three matrices tested revealed no deterioration of the matrices and only insignificant plugging.

Julian

TABLE OF CONTENTS

	<u>Page</u>
Foreword	1
Abstract	ii
Table of Contents	iii
List of Tables and Figures	iv
1.0 SUMMARY	1
2.0 INTRODUCTION	2
3.0 EXPERIMENTAL OPERATIONS	5
3.1 Cesium Vaporizer Test Rig	5
3.2 Matrix Evaluation Techniques	8
3.2.1 Radiography	8
3.2.2 Spectrographic and Metallographic Examinations	8
4.0 EXPERIMENTAL RESULTS	10
4.1 Radiography	10
4.2 Cesium Analyses	10
4.3 Spectrographic Analyses of Matrices	10
4.4 Metallographic Analyses of Matrices	17
5.0 DISCUSSION OF RESULTS	22
5.1 X-ray Evaluations	23
5.2 Cesium Condensate Analysis	23
5.3 Spectrographic Evaluations of Matrix Sections	26
5.4 Metallographic Evaluations	29
5.5 Relation of Test Results to Ion Engine Operation	30
6.0 CONCLUSIONS	31

LIST OF TABLES AND FIGURES

		<u>Page</u>
TABLE 4-1	Cesium Analysis	14
TABLE 4-2	Semi Quantitative Analyses of Matrices	15
TABLE 4-3	347 Stainless Steel Composition of Matrix Sections .	16
TABLE 4-4	Cesium and Sodium Concentrations in Matrix Sections .	16
TABLE 5-1	Iron, Chromium, and Nickel Ratios in 2100 Hour Test Matrix Sections	26
FIGURE 3-1	Cesium Vaporizer Test Rig	6
FIGURE 3-2	Matrix Sectioning Diagram	9
FIGURE 4-1	X-ray Photographs of Matrix Number One	11
FIGURE 4-2	X-ray Photographs of Matrix Number Two	12
FIGURE 4-3	X-ray Photographs of Matrix Number Three	13
FIGURE 4-4	Micro and Macro Cross Section of Control Matrix . . .	18
FIGURE 4-5	Macrographs of Matrices Exposed to Cesium Vapor . . .	19
FIGURE 4-6	Photomicrographs of Typical Microstructures of Matrices Exposed to Cesium Vapor	20,21

POROUS MATRIX PHASE SEPARATOR EVALUATION

By

Keith M. Montgomery

Thompson Ramo Wooldridge Inc.

1.0 SUMMARY

A research program to build and test a porous matrix phase separator applicable for an ion engine propulsion system was conducted by TRW from March 1963 to July 1964. The possibility arises of matrix plugging and corrosion deterioration resulting from continuous long term propellant vaporization within the matrix. To experimentally evaluate these possibilities a test rig containing three similar matrices, that vaporized cesium metal for 2100 hours at 800°F and 0.5 psi, was designed, built, and operated. At each 700 hour test interval the matrices were x-rayed to determine the extent of plug formations. At the conclusion of the test metallographic and spectrographic analyses of the matrices were conducted.

The x-ray photographs showed no change in the matrix structure or plugging in the matrices as a result of the continuous cesium boiling. Sodium, an impurity in the supply cesium, was found in small quantities in each of the matrices. Sodium and no other impurity in the cesium (that was not common to the 347 stainless steel matrix) was found in concentrations greater than 1000 ppm in the liquid inlet, meniscus zone, or vapor exit portions of the matrix. The degree of plugging was not significantly great to cause any alteration of the cesium boiling rate or detract from its normal intended operation in an ion engine feed system. Corrosion and erosion of the matrices resulting from leaching of stainless steel elements was not observed.

Metallographic examinations showed that the grain boundaries in the type 347 stainless steel matrix were not preferentially attacked by the cesium. Also, there was no change in the crystalline structure of the matrix.

2.0 INTRODUCTION

On March 18, 1963, Contract NAS3-2518 was awarded to TRW for evaluation of a porous matrix phase separator. This separator is one component of a cesium propellant feed system for an ion propulsion engine. It was intended that the investigation determine the matrix plugging and deterioration characteristics that may result from long term continuous vaporization of cesium.

The interest in this study stems from the possibility of using a porous vaporizer for stable and complete phase separation in a zero-gravity environment. The performance of these matrices over long periods of unattended operation had not been evaluated previously. There exists the possibility that matrices will be plugged as a result of concentration of the cesium impurities at the boiling liquid vapor interface. Erosion and corrosion of the matrices due to continual exposure to the alkali metal vapor was considered to be another possibility.

Impurities normally contained in cesium originate from the native mineral source, from the method of processing and reducing the cesium compound, and from container and system materials during final application. In processing, a purified cesium chloride or bromide is reduced by an active metal (usually Ca or Ti). Under vacuum, the liberated cesium metal is distilled from the reaction mixture. The active metal reductant also functions as a nitrogen or oxygen getter in the reaction chamber atmosphere. Common impurities reduced and distilled with the cesium are the alkali metals, Rb, K, Na, and Li. It may happen that improper control of temperatures or poor design of the apparatus will permit small amounts of the reductant metal to be entrained with the distilling cesium. The concentration of oxide and nitride in the product is much dependent on the quality of the vacuum system, the absence of system leaks and the care exercised in degassing the entire apparatus before reduction and distillation are begun.

During operation, impurities may be acquired as a consequence of leaks of atmospheric gases and of direct attack on the fuel system. As a result of protracted storage in metal systems, cesium tends to dissolve components (Ni, Fe, Cr, Mn, etc.) from containment alloys. However, direct solution is a less aggravated form of attack than the preferential leaching of carbides deposited in steel grain boundaries. This mode of attack appears to be the most critical form

for the alkalis and will contribute significantly to the impurity levels in the flowing media.

It is expected that the cesium liquid entering the porous vaporizer will carry with it the following primary impurities:

Rubidium	Nickel	Sodium
Potassium	Chromium	Iron
Lithium	Titanium	Calcium

As well as oxides, nitrides, and azides.

Impurities are not generally present in elemental form, but rather will appear as intermetallic compounds or solutions. Their activity at the boiling interface was of prime concern during the program. Some vaporization of all impurities should occur. This vaporization is the result of the tendency of all the components of the liquid phase to come into equilibrium with identical components of the vapor phase.

A further loss of impurities from the boiling liquid is caused by physical carryover due to the vaporizing process. This carryover is not to be confused with gross liquid carryover; rather it constitutes a physical removal of molecules of impurities due to thermal and physical agitation at the boiling interface. Some impurity constituents remain behind unvaporized and slowly build up in concentration at the interface. It is this build up which eventually may cause pore obstructions.

Another possible vaporizer failure may arise from direct chemical attack on the sintered structure. As mentioned, selective solution of carbides precipitated at grain boundaries may be the dominating alkali corrosion reaction in stainless steels. Solution and alloying effects are comparatively negligible. The presence of oxides and other impurities generally accelerate corrosion. The use of a type 347 stainless steel was employed in the porous matrices to reduce the extent of chemical attack since the concentration of precipitated carbides is minimized in this series.

The study of the above mentioned factors which may contribute to matrix plugging of deterioration was accomplished with a test rig designed and built for the

continuous vaporization of cesium with three matrices. These matrices were x-rayed before, during and at the conclusion of the tests. Spectrographic and metallographic analyses of the matrices were made following the completion of the tests.

3.0 EXPERIMENTAL OPERATIONS

The description of the experimental efforts of this program is divided into two major subsections. These are:

1. The test rig and its operations.
2. Analytical techniques for matrix evaluations.

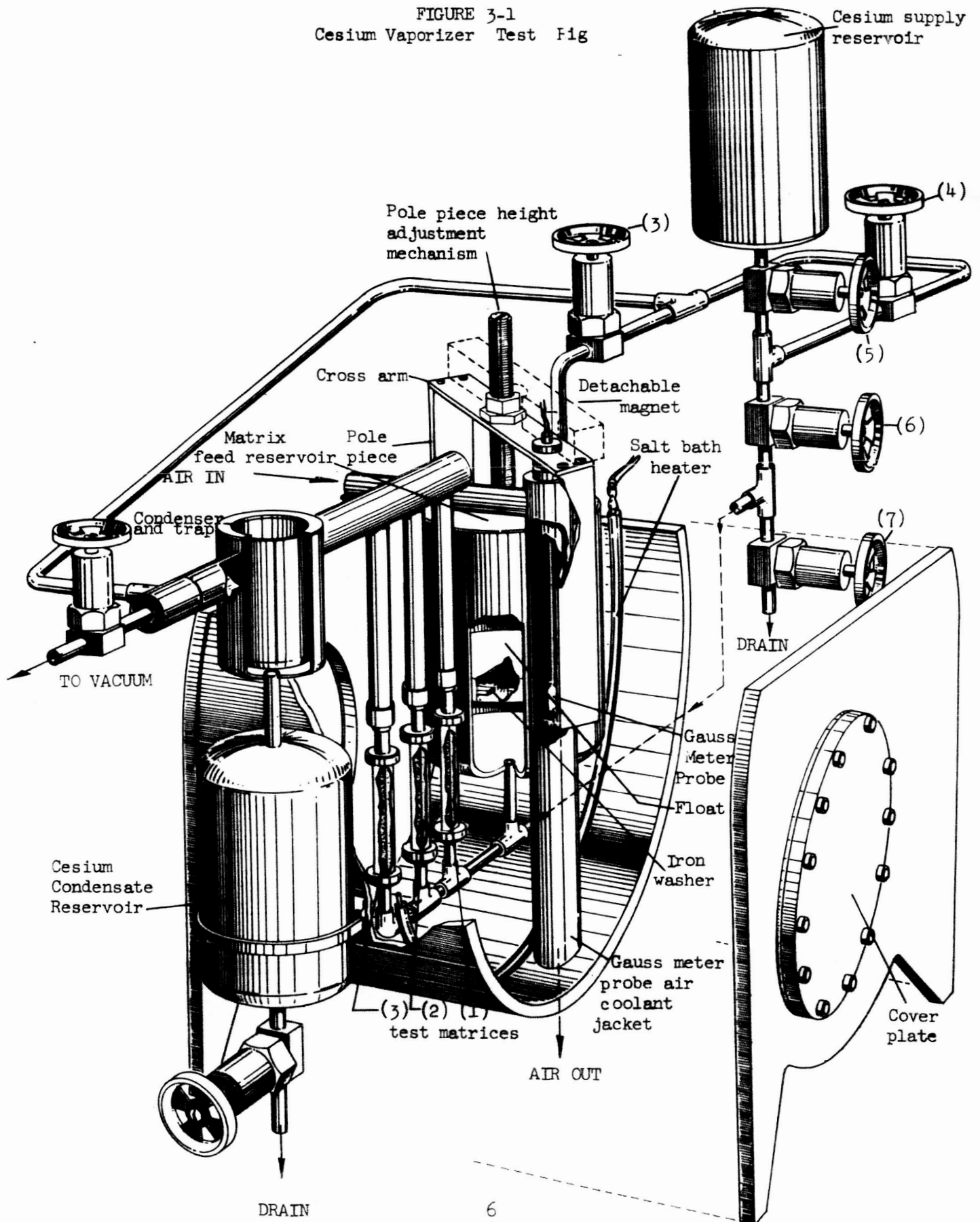
3.1 Cesium Vaporizer Test Rig

An artist's sketch of the test rig used in the vaporizer evaluation experiments is shown in Figure 3-1. All components were fabricated from either 347 or 316 stainless steel. The flow control valves were Hoke type TY-445 bellows-sealed globe valves. The matrices and the matrix feed reservoir were heated by being immersed in a salt bath mixture of potassium and sodium nitrate. Two tubular 150 watt heaters maintained this salt bath at $800 \pm 10^\circ\text{F}$.

Fabrication techniques used in preparation of the matrices were aimed primarily at enhancing matrix evaluations by x-ray studies. The sheath of each matrix was fabricated from a type 316 stainless steel solid rod. A 1/4 inch square hole four inches long was Eloxed through the center of the rod. Next, type 347 stainless steel powder was packed into the square center void. On sintering, a porous structure of a one-quarter inch square cross section having an average porosity of 50 per cent was produced. Since the most distinct x-ray photograph of the matrix sample could be obtained by minimizing the sheath thickness, the final stage in specimen preparation was the machining of the sheath walls to within 0.020 inches of the porous center section.

A magnetic liquid level detection system was used to measure the feed inventory of cesium in the matrix feed reservoir during the course of the vaporizer evaluation tests. A float containing an iron washer was inserted into the matrix feed reservoir. The position of the iron washer corresponded to a definite cesium liquid level within the reservoir. By means of a magnet pole piece (immersed in the salt bath) the position of the iron washer could be found with the aid of a gaussmeter and gauss probe. Moving the pole piece up and down, the gauss probe (which is permanently attached to the cross arm of the pole piece) cuts the magnetic lines of force between the iron washer and the magnet on the cross arm. The position of the gauss probe that corresponds

FIGURE 3-1
Cesium Vaporizer Test Fig



to the maximum magnetic field flux density also corresponds to the position of the iron washer or the height of the liquid cesium in the matrix feed reservoir.

A Radio Frequency Laboratory Model 1295-A gaussmeter and a model 1295 probe was used for the liquid level measurements. A fixed orifice downstream of each matrix regulated the cesium flow through the matrices. The diameter of these orifices was sized to permit a total mass flow rate of one pound of cesium to be vaporized and condensed over a 700 hour test period.

To operate the test rig the unit was first evacuated to a pressure of 50 microns or less. Next the salt bath was brought up to the operating temperature ($800 \pm 10^\circ\text{F}$), and kept there during the course of the entire experiment. Once temperature equilibrium among all the test rig components had been attained valves 2, 3, and 4 (Figure 3-1) were closed and the gaussmeter probe was positioned at a height midway between the inlet and exit ends of the matrices. Valve 5 was then opened and cesium was allowed to drain from the supply reservoir into the matrix feed reservoir. When the iron ring in the float rose to the gaussmeter probe height the cesium supply was shut off by first closing valve 5 and then valve 6. Since each of the vaporizers was located in parallel with the feed reservoir, the height of the liquid cesium in this tank was approximately the same as that determined by the height of liquid cesium in the test matrices.

Three matrices, rather than one were used in the experiment for comparison. As cesium was vaporized from the matrices the liquid level in the feed reservoir dropped. When this level dropped one inch below its initial setting valves 5 and 6 were again re-opened to admit more cesium to the feed reservoir and re-establish the initial liquid level. By operating in this manner the meniscus zone in each matrix was restricted to a limited region.

One pound of cesium was consumed by the vaporization process over a 700 hour test period. Following consumption of each pound of cesium, the system was temporarily shut down, the salt was drained from the bath, and x-rays were taken of the matrices. A fresh supply of cesium was then put into the test rig, the system was again brought up to temperature, and the vaporizer experiment was continued. This procedure was repeated three times resulting in a matrix test of 2100 hours while vaporizing a total of 3 pounds of cesium through the test rig.

In designing the test rig, it was intended that the operating parameters (system temperature and pressure) should simulate the expected working conditions of a feed system for an ion propulsion engine. The geometrical dimensions of the matrices such as particle size and shape were chosen on the basis of obtaining a unit that could be easily x-rayed, be uniform in consistency, and still conform to a bench type laboratory test rig.

The cesium used in the experiments was high grade commercially available material that had an average analyses of 99.9 per cent cesium. Cesium of this purity is used in ion engine operations.

3.2 Matrix Evaluation Techniques

During and at the conclusion of the vaporization studies, the matrices were evaluated by three different means (i.e., x-ray analysis, spectrographic analysis, and metallographic analysis). The procedure used in each of these methods is described below.

3.2.1 Radiography

In developing the x-ray process for these matrix studies it was determined that a 150 kv tube potential was ideal for the test series. This relatively low tube potential produces radiation at an energy level which results in a film image of the matrices having good contrast and detail. In addition, the x-ray tube used for this series had a characteristic anode focal spot of 0.3 mm. (The average industrial x-ray tube has a focal spot dimension in the order of 5 mm). By using this small focal spot the detail of the image is extremely sharp and there is no distortion present due to poor x-ray focusing. It is common practice to place an aluminum screen on both sides of the film during exposure. The use of such screens cuts down on scatter radiation and increases the image detail. With the square matrix geometry and the above tube specifications, an aluminum screen was not necessary for precise definition. The film is an extra fine grain material which again further optimizes contrast and sensitivity.

3.2.2 Spectrographic and Metallographic Examinations

Spectrographic and metallographic examinations were conducted after conclusion of the vaporizer studies to evaluate the condition of the matrices

at the liquid inlet, meniscus zone, and vapor exit. Each matrix was sectioned according to the accompanying diagram.

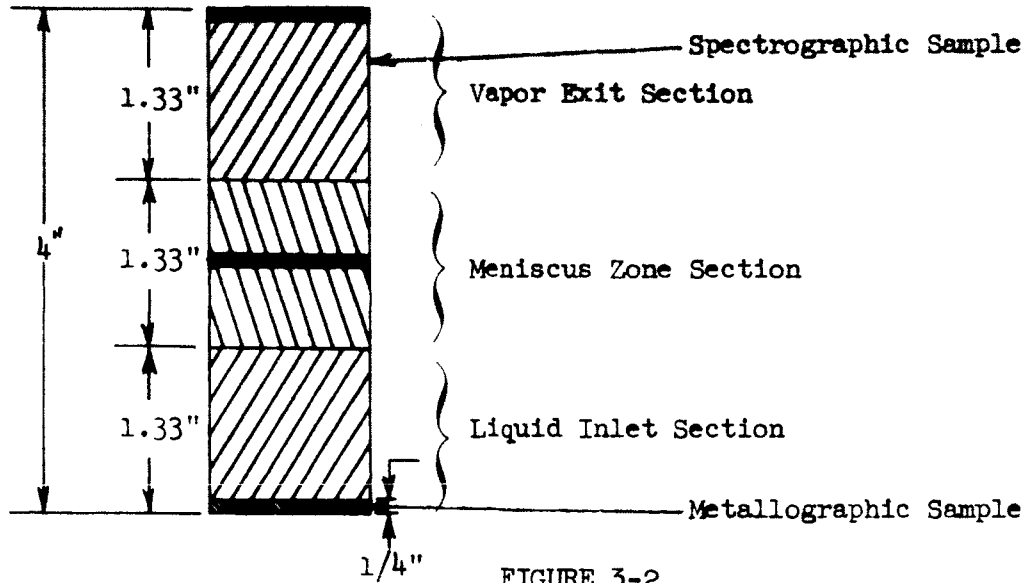


FIGURE 3-2

Matrix Sectioning Diagram

4.0 EXPERIMENTAL RESULTS

4.1 Radiography

X-ray photographs, Figures 4-1, -2, -3, indicate the condition of the matrices after different periods of exposure to cesium. The x-ray conditions in each case were:

Tube Potential - - - - 150 kv at 10 milliamps

Exposure Time - - - - 45 seconds

Fixed Focal Distance - 48 inches

Anode Focal Spot - - - 0.3 millimeters

Film - - - - - Eastman Kodak Type M

4.2 Cesium Analyses

The impurity levels found in the initial untested cesium and the cesium condensate collected during the 1400 to 2100 hour test period are shown in Table 4-1. All cation analyses employed spectrographic techniques. The oxygen analyses were performed with neutron activation techniques.

As a means of substantiating the oxygen concentration measurements the purity of the cesium condensate was also measured by the freezing point method. The freezing point of the untested cesium was 28.50°C and it became 28.08°C as a result of the 1400 to 2100 hour test. Assuming this impurity build up was due only to oxygen, the oxygen level of the cesium condensate is estimated by this means to be 0.0200 per cent.

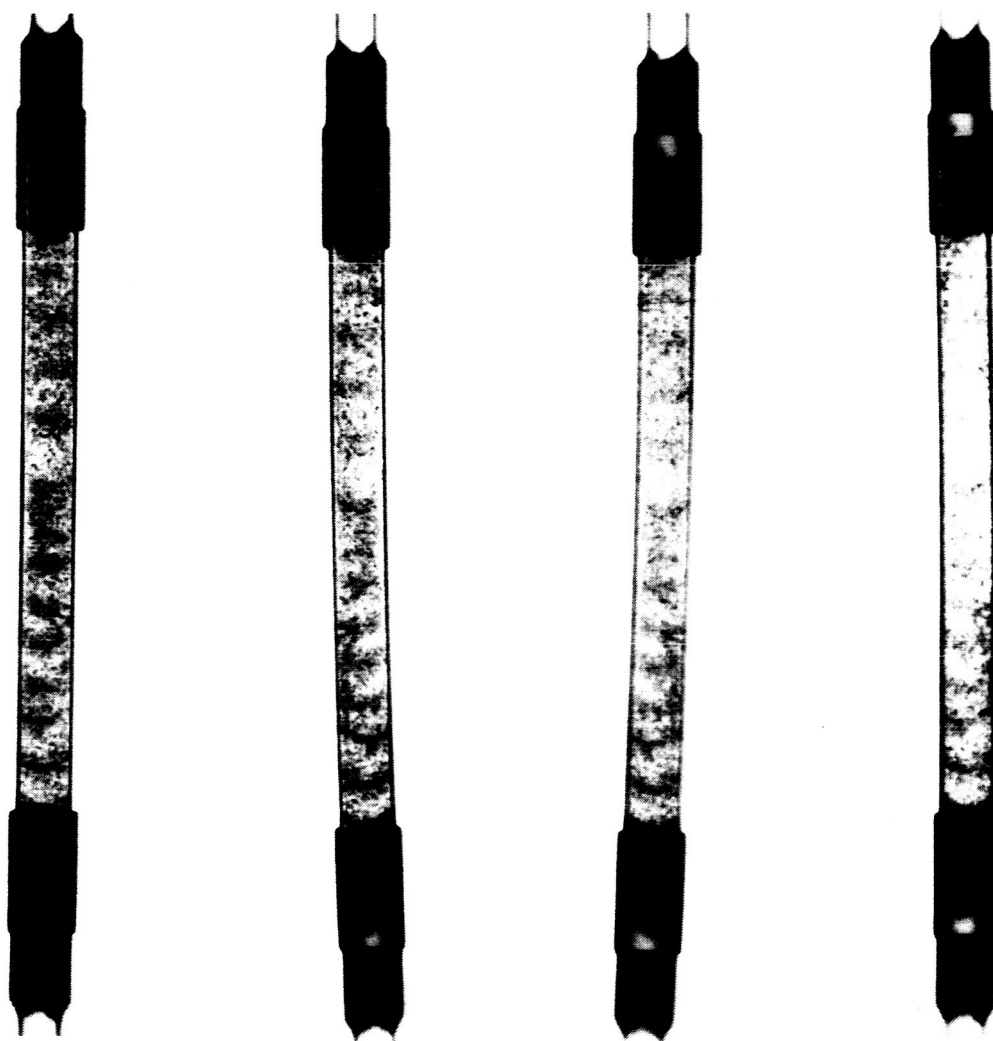
4.3 Spectrographic Analyses of Matrices

The results of the spectrographic analyses of the matrices are presented in Tables 4-2, -3, and -4. These analyses were performed at the conclusion of 2100 hours of test time (vaporization at 800°F). Table 4-2 shows the results of a semi quantitative analysis of the liquid inlet, meniscus zone, and vapor exit from each of the three matrices as well as a similar analyses from a control (unexposed) matrix.

In Table 4-3 is shown the results of the quantitative analyses of the primary type 347 stainless steel elements found in each of the matrix sections as well as the control matrix.

FIGURE 4-1

X-RAY PHOTOGRAPHS OF MATRIX NUMBER ONE



Matrix prior
to cesium exposure

Matrix after
700 hours of
cesium exposure

Matrix after
1400 hours of
cesium exposure

Matrix after
2100 hours of
cesium exposure

FIGURE 4-2

X-RAY PHOTOGRAPHS OF MATRIX NUMBER TWO



Matrix prior
to cesium exposure



Matrix after
700 hours of
cesium exposure



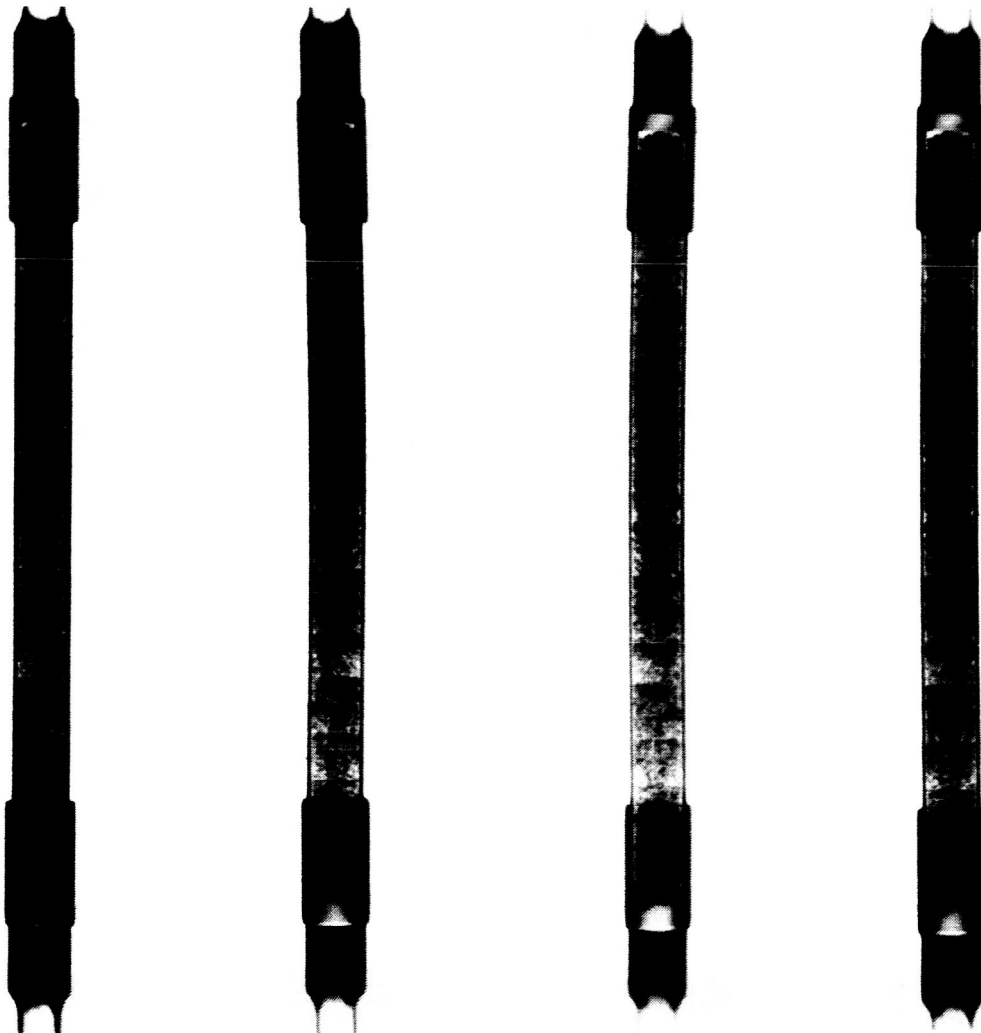
Matrix after
1400 hours of
cesium exposure



Matrix after
2100 hours of
cesium exposure

FIGURE 4-3

X-RAY PHOTOGRAPHS OF MATRIX NUMBER THREE



Matrix prior
to cesium exposure

Matrix after
700 hours of
cesium exposure

Matrix after
1400 hours of
cesium exposure

Matrix after
2100 hours of
cesium exposure

TABLE 4-1

Cesium Analysis

<u>Element</u>	<u>Initial Cesium</u>	<u>Cesium Condensate</u> (From 1400 to 2100 hour test period)
Al	<0.0002%	0.0002%
Ba	0.0008	<0.0008
B	<0.0018	<0.0016
Ca	0.0018	0.0035
Cu	<0.0002	<0.0002
Cr	<0.0002	<0.0002
Fe	<0.0014	0.0003
Mg	0.0005	0.0002
Mn	0.0005	<0.0002
Ni	<0.0002	0.0002
Pb	<0.0002	<0.0002
Si	<0.0004	<0.0003
Sn	0.0008	<0.0008
Sr	<0.0002	<0.0002
Li	0.0018	<0.0016
Ti	<0.0002	<0.0002
Tl	<0.0002	<0.0002
Na	<0.0016	0.0008
K	0.0007	0.0006
Rb	0.0035	0.0024
O ₂	0.0011	0.0404

TABLE 4-2

SEMI QUANTITATIVE OF ANALYSES OF MATRICES

ELEMENT	CONTROL	Matrix No. 1			Matrix No. 2			Matrix No. 3		
		VAPOR EXIT	MENISCUS ZONE	LIQUID INLET	VAPOR EXIT	MENISCUS ZONE	LIQUID INLET	VAPOR EXIT	MENISCUS ZONE	LIQUID INLET
Al	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Ti	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Cu	.01 to .10%	.01 to .10%	.01 to .10%	.01 to .10%	.01 to .10%	.01 to .10%	.01 to .10%	.01 to .10%	.01 to .10%	.01 to .10%
V	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05
Mg	Not detected	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Mo	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05	.005 to .05
Co	.01 to .10	.01 to .10	.01 to .10	.01 to .10	.01 to .10	.01 to .10	.01 to .10	.01 to .10	.01 to .10	.01 to .10
Ba	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Na	Not detected	.01 to .02	.05 to .1	.05 to .1	.2 to .4	.2 to .4	.08 to .2	.03 to .05	.08 to .2	.05 to .1
K	Not detected	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Li	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Cs	Not detected	1-10	1-10	1-10	1-10	1-10	1-10	1-10	1-10	1-10

The following elements were analyzed for but not found in either the control or test matrices:

Rb, Cd, Ca, Bi, Zr, Pb, Sn, Ag, Sb, As, B, Zn, Sr, and W

TABLE 4-3

347 STAINLESS STEEL COMPOSITION OF MATRIX SECTIONS

ELEMENT	Matrix No. 1				Matrix No. 2				Matrix No. 3			
	CONTROL	VAPOR EXIT	MENISCUS ZONE	LIQUID INLET	VAPOR EXIT	MENISCUS ZONE	LIQUID INLET	VAPOR EXIT	MENISCUS ZONE	LIQUID INLET	VAPOR EXIT	LIQUID INLET
Cr	18.37%	17.63	17.38	18.99	19.12	18.16	18.64	17.57	19.05	17.91		
Mn	0.19	0.19	0.19	0.19	0.19	0.19	0.19	0.19	0.19	0.19		
Ni	10.83	11.23	10.31	11.13	10.82	10.60	10.86	10.10	11.07	10.66		
Cb	0.72	0.73	0.72	0.73	0.72	0.73	0.73	0.71	0.73	0.73		
Si	0.89	0.88	0.89	0.89	0.89	0.89	0.88	0.89	0.89	0.89		
Fe	68.9	67.3	67.4	63.8	62.9	64.1	63.2	64.8	62.2	64.0		

TABLE 4-4

CESIUM AND SODIUM CONCENTRATIONS IN MATRIX SECTIONS

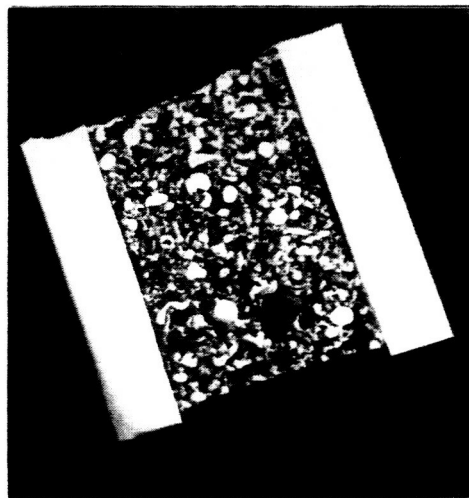
ELEMENT	Matrix No. 1				Matrix No. 2				Matrix No. 3			
	CONTROL	VAPOR EXIT	MENISCUS ZONE	LIQUID INLET	VAPOR EXIT	MENISCUS ZONE	LIQUID INLET	VAPOR EXIT	MENISCUS ZONE	LIQUID INLET	VAPOR EXIT	LIQUID INLET
Cs	Not detected	1.94%	2.93	4.11	4.96	4.94	5.35	5.58	5.64	5.47		
Na	Not detected	0.015%	0.070	0.080	0.28	0.31	0.10	0.045	0.11	0.090		

All other elements from Table 4-2 that were present in concentrations greater than 1000 ppm (0.10 per cent) were quantitatively analyzed and are reported in Table 4-4.

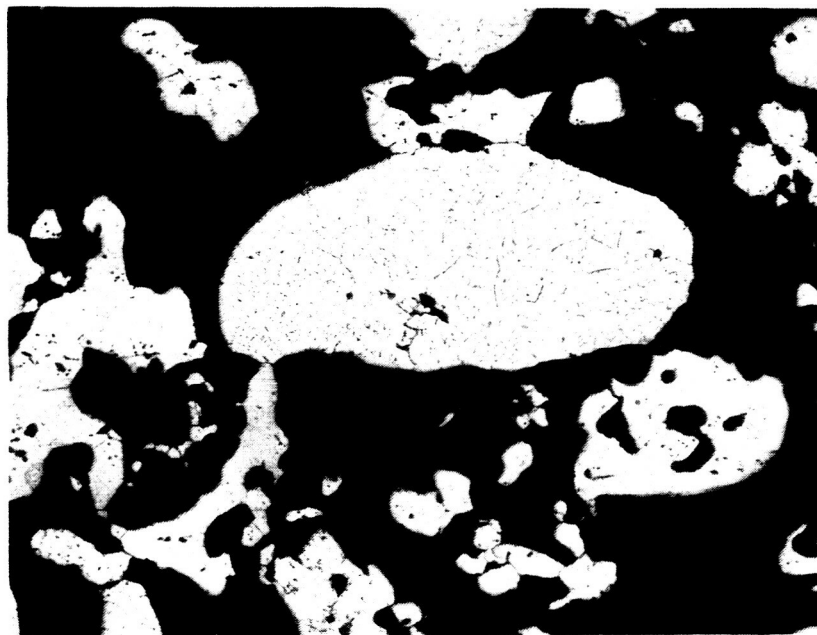
4.4 Metallographic Analyses of Matrices

Macro and micro cross sections of the control matrix and tested matrix sections are shown in Figures 4-4, -5, and -6. The micro cross sections shown in Figure 4-6 were typical of the cross sections found throughout the entire body of all three of the matrices.

07879-1



3X



A177

100X

HF-H₂SO₄-HNO₃ ETCH

FIGURE 4-4

MICRO AND MACRO CROSS SECTION OF CONTROL MATRIX

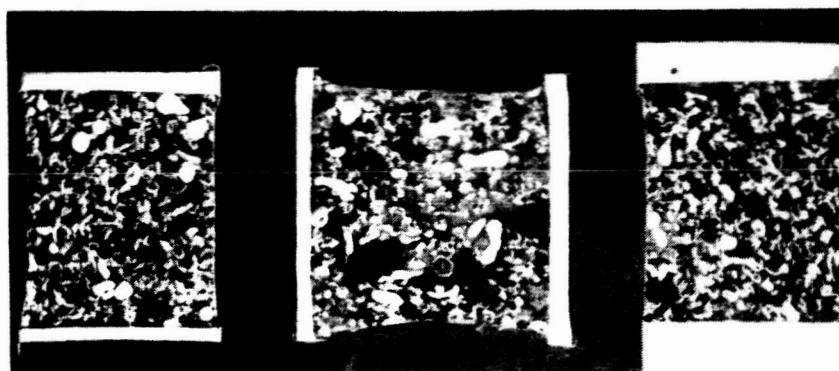


Liquid Inlet
07879-3

Meniscus Zone

Vapor Exit

MATRIX NUMBER 1

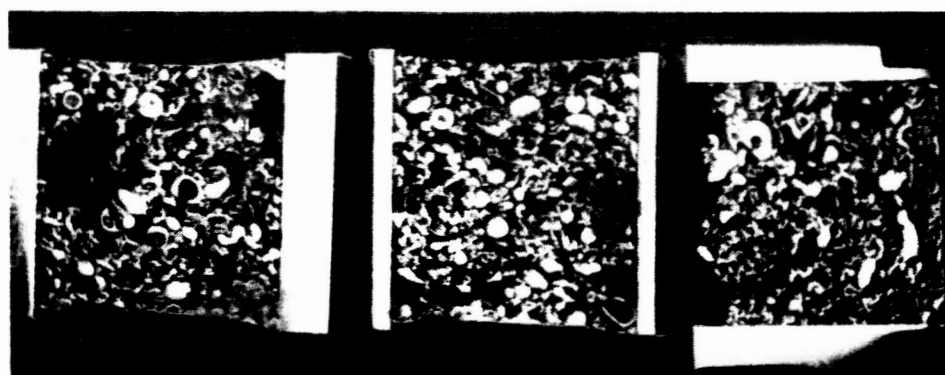


Liquid Inlet
07879-2

Meniscus Zone

Vapor Exit

MATRIX NUMBER 2



Liquid Inlet
07879-4

Meniscus Zone

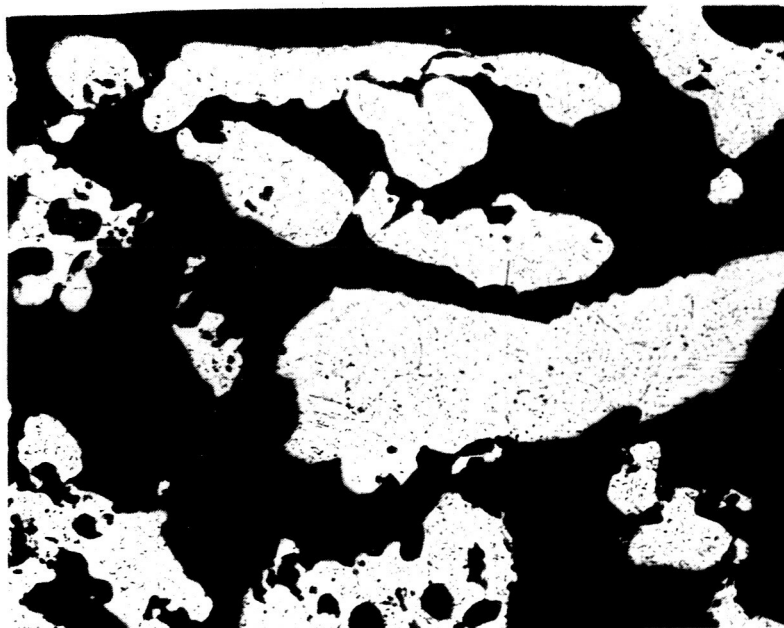
Vapor Exit

MATRIX NUMBER 3

FIGURE 4-5

MACROGRAPHS OF MATRICES EXPOSED TO CESIUM VAPOR

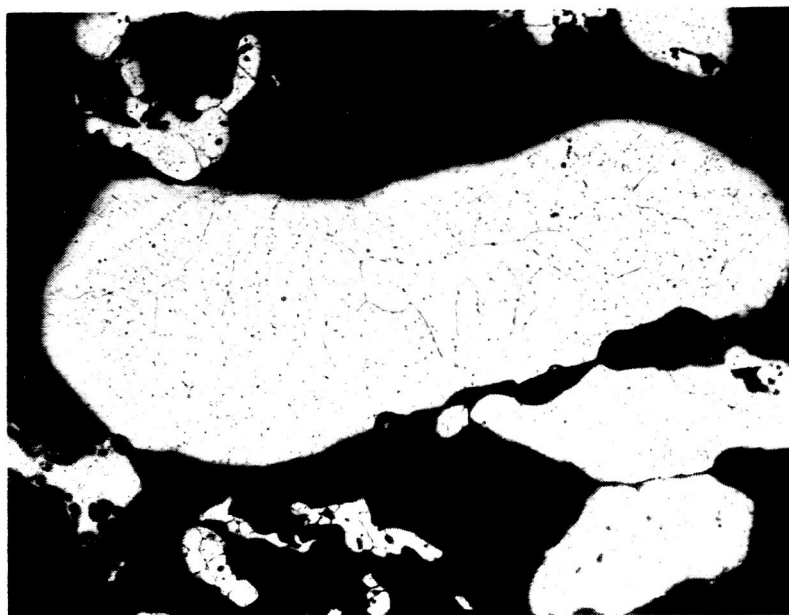
3X



A 178

100X

MENISCUS ZONE OF MATRIX No. 1



A 179

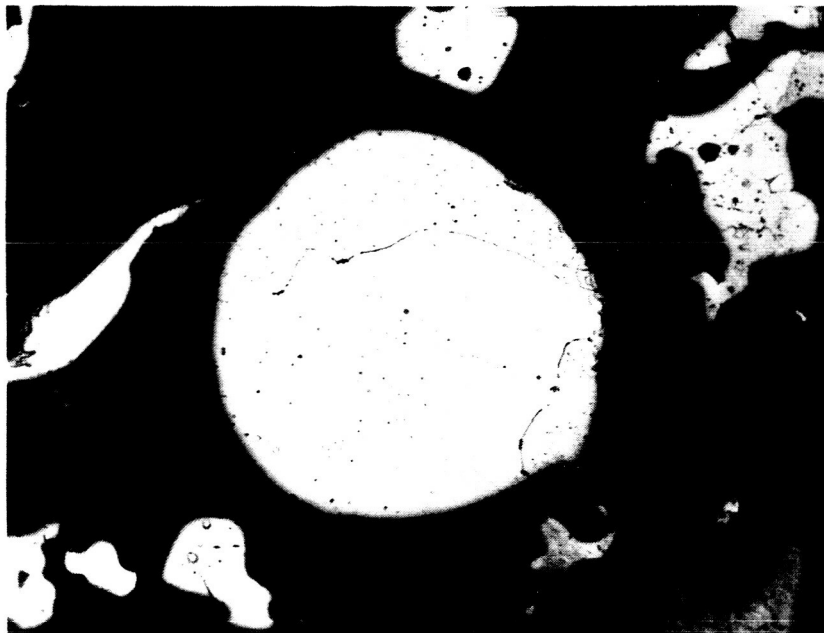
100X

MENISCUS ZONE OF MATRIX No. 2

HF-H₂SO₄-HNO₃ ETCH

FIGURE 4-6

PHOTOMICROGRAPHS OF TYPICAL MICROSTRUCTURES OF MATRICES
EXPOSED TO CESIUM VAPOR



A 180

100X

MENISCUS ZONE OF MATRIX No. 3

FIGURE 4-6 (Continued)

5.0 DISCUSSION OF RESULTS

Over long periods of continuous boiling, impurities in the propellant will tend to concentrate in the matrix, and cause plugging. Also chemical corrosion and mechanical erosion will deteriorate the vaporizer. The experiment was undertaken specifically to supply more information in these areas. For this reason no attempts were made to measure pressure drop across the matrices, or to simulate any boiling conditions that might be experienced in zero gravity.

The experiment was designed with the intent of approaching as near as possible actual operating conditions expected to be experienced by a feed system of an ion propulsion rocket. The test temperature (800°F) was chosen partially because it meets this requirement and partially because it is high enough to accelerate any possible detrimental effects resulting from corrosion or erosion.

The cross sectional dimensions of the matrices were chosen on the basis of optimizing the clarity of the x-ray photographs. The length of the matrix (4 inches) approaches the maximum length that the supplier could make and still furnish uniform constituency throughout the vaporizer's entirety.

Since experiments of this nature had not been previously performed, three similar matrices were tested simultaneously in the experiments. In the present state of development, results obtained in this manner should be more significant than results obtained from three physically different matrices tested under the same conditions.

The following criteria were used in evaluating the results presented in Section 4:

1. Formation of possible vaporizer plugs resulting from concentration of basic impurities in the supply cesium.
2. Formation of possible vaporizer plugs resulting from redistribution of stainless steel elements in the matrices due to continual exposure to cesium metal.
3. Formation of possible vaporizer plugs resulting from impurity concentration at the liquid-vapor interface of the boiling cesium.

4. Loss of stainless steel elements from the matrices due to a leaching phenomena caused by cesium.
5. Carry over of the impurities and contaminants due to entrainment from the boiling cesium.

5.1 X-ray Evaluations

As evidenced in Figures 4-1, 4-2, and 4-3, the matrices have the same appearance both before and after 2100 hours exposure to cesium. The x-ray photographs show no change in matrix structure or build up of cesium impurities during the course of the 2100 hour experiment. Solely by the use of x-ray analysis a meniscus zone as discernible by impurity build up could not be determined.

The apparent granular structure of the matrices is due to a random alignment of void areas in the viewing direction. If the metallic particles were uniformly packed the distinctive dark and light areas in the x-ray photographs would not be as apparent. The macro and micrographs shown in Figure 4-4 and 4-5 also indicate the non-uniformity of the matrix makeup.

5.2 Cesium Condensate Analysis

The data shown in Table 4-1 are the results of an analysis of the condensate of a one pound sample of cesium metal that was vaporized and condensed over the 1400 hour to 2100 hour test period.

Within experimental error the impurity level for all tested elements, with the exception of Ca, Na, and Rb, did not change during the vaporization process. The calcium concentration increased from 0.0018 per cent to 0.0035 per cent. The sodium concentration initially less than 0.0016 per cent was finally 0.0008 per cent. During the test the rubidium level decreased from 0.0035 per cent to 0.0024 per cent. The reason for the increased calcium concentration by almost a factor of two is not readily apparent. The boiling point at one atmosphere for calcium is about 2270°F. At the system operating temperature (800°F), it is not probable that calcium was being vaporized even at the reduced operating pressure which was approximately 0.5 psia (the vapor pressure of Cs metal at 800°F).

An unlikely explanation for the increased calcium level in the cesium condensate could be accredited to calcium build up at the boiling interface and then periodic carry over due to a surging phenomena of the boiling cesium. This could occur as the result of the calcium being concentrated in the matrices during the vaporization of the two one-pound lots of cesium tested over the first 1400 hours of the experiment and then being carried over by entrainment into the cesium condensate during the 1400 to 2100 hour portion of the experiment. However, calcium was not detected in any of the matrices either before or after the tests. Therefore, it is not probable that the element was being concentrated by means of a liquid carry over.

Initially the Na level in the cesium (Reference Table 4-1) was less than 0.0016 per cent while the condensate contained 0.0008 per cent sodium. The improvement in the preciseness in these two numbers reflects the improvement in analyses used in the determinations. Over the time interval in which the two sets of analyses were made the sensitivity of the sodium analysis techniques was improved to the extent that measurements in the range of 8 ppm could be determined. At the time the initial cesium was supplied these analyses techniques were not available.

Sodium concentration in the matrices (considering that the untested units had no sodium) can be explained by observing the boiling point of the element. At a pressure of 10 torr (which is slightly lower than the system operating pressure of 26 torr) the boiling point of the alkali metals are:

Lithium	1634°F	(1)
Sodium	1018°F	
Potassium	829°F	
Rubidium	729°F	

Because the operating temperature of the system was 800°F, the trend of this data indicates that sodium and lithium would be concentrated at the boiling interface while potassium and rubidium will be boiled off along with the cesium.

(1) "Liquid Metals Handbook", Atomic Energy Commission, Department of the Navy, Washington, D. C., June 1952.

Actually some lithium may have been separated from the cesium supply during vaporization. The lithium level in the condensate was less than 0.0016 per cent while the initial lithium concentration in the cesium was 0.0018 per cent.

Because of the sodium content found in the matrices it is expected that the sodium concentration in the cesium effluent would be of smaller magnitude than the sodium concentration in the supply cesium.

During the 1400 to 2100 hour vaporization period the rubidium concentration in the cesium changed from 0.0035 per cent in the initial material to 0.0024 per cent in the collected condensate. This indicates that most of the rubidium was being vaporized with the cesium and thus is not likely to cause plugging within a matrix vaporizing system. This was further substantiated by the spectrographic analyses of the matrices (Table 4-2) for rubidium was not detected in any of the matrix sections.

When evaluating the matrix performance characteristics over long periods of operation the possibility of leaching of the stainless steel elements by the cesium becomes of interest. In considering only the analysis of the cesium condensate, the leaching phenomena, if it did occur, was not apparent. The iron, nickel, and chromium levels in the condensate cesium samples were not significantly different from these levels in the initial cesium. This leaching phenomena will be discussed further in Section 5.3.

The oxygen content of the cesium condensate (collected during the 1400 to 2100 hour test period) was determined to be approximately 400 ppm by neutron activation measurements and 200 ppm by freezing point depression techniques. The magnitude of difference between these two numbers illustrates the difficulty in precise measurements of low concentrations of oxygen in cesium. The discrepancy between the two sets of results points out the need for further improvement in means for analyzing oxygen in cesium.

Since the oxygen level in the cesium effluent was in the range of 200 to 400 ppm, there was no substantial air leakage from the outside environment to the interior of the test rig during the course of experimentation. The source of the additional oxygen was either residual oxygen entrapped from interior portions of the test rig, minute accumulative air leaks, or handling and transfer techniques.

5.3 Spectrographic Evaluations of Matrix Sections

The data shown in Tables 4-2, 4-3, and 4-4 are the results of spectrographic analyses on various sections of the three test matrices.

Of all the metals listed in Table 4-2 only cesium and sodium were found to be present in concentrations greater than 1000 ppm. Furthermore with the exception of the alkalies, there appeared to be no segregation or no other material build up in any one location of the matrices. A single layer of impurity build up resulting from concentration of cesium impurities in the boiling process was not observed. This is in keeping with the finding of the x-ray studies, (Figures 4-1, 4-2, and 4-3). Here as well as in the spectrographic data of Table 4-2, the consistency of the matrices appeared to remain for the most part unaltered during the course of the vaporizer studies.

As shown by the data in Table 4-3, there are some variances from the control matrix in the iron, nickel, and chromium contents of each of the tested matrices, while this was not so for the manganese, columbium, and silicon concentrations.

Since iron is the major constituent in the matrices, the reason for the drop in iron concentration from the control sample is attributed to the two to five per cent cesium levels found in the tested vaporizers (see Table 4-4). The interesting observation to note is the iron to chromium and iron to nickel ratios found in the various matrix sections. The results of these calculations are presented in Table 5-1.

TABLE 5-1

Iron, Chromium, and Nickel Ratios in 2100 Hour Test Matrix Sections

Ratio	Control	<u>Matrix No. 1</u>			<u>Matrix No. 2</u>			<u>Matrix No. 3</u>		
		Vapor Exit	Meniscus Zone	Liquid Inlet	Vapor Exit	Meniscus Zone	Liquid Inlet	Vapor Exit	Meniscus Zone	Liquid Inlet
Fe/Cr	3.7	3.8	3.9	3.3	3.3	3.5	3.4	3.6	3.3	3.6
Fe/Ni	6.4	6.0	6.6	5.6	5.8	6.1	5.8	6.4	5.6	6.0
Cr/Ni	1.7	1.6	1.7	1.7	1.8	1.7	1.7	1.8	1.7	1.7

These ratios indicate that there is no significant variation in either the iron-to-chromium or iron-to-nickel ratio along the entire length of the matrix.

If metal migration or leaching had occurred, it would have taken place by one of two mechanisms.

1. Group transport of all stainless steel constituents.
2. Selective leaching of individual stainless steel components.

If all the elements in the stainless steel were being leached at a constant rate from one section of the matrix to another, examination of the individual elements ratios or percentage concentrations would not substantiate the leaching occurrence. However, on the basis of the test data in Table 4-3, there is no reason to believe that this group transport occurred.

The scatter of the ratios in Table 5-1 is hardly more than would be expected in experimental analyses errors. By considering the chromium to nickel ratios (the third column in Table 5-1) the consistency of the data was even more apparent. Therefore, it was concluded that selective leaching of individual elements within the stainless steel did not occur.

To verify that the spectrographic analyses reported all the major constituents in the matrices, the sum of the concentrations listed in Tables 4-3 and 4-4, for any singular vaporizer section, should be equal to unity. This was true within one per cent and is attributed to accumulative experimental error and to trace concentrations of elements not listed in Table 4-3 or 4-4.

The data contained in Table 4-4 shows the quantitative analysis of all elements found in the matrices in concentrations greater than 1000 ppm that were not constituents of the type 347 stainless steel.

The reason for performing this analysis was to determine the concentration of large impurity build up resulting from the continuous boiling of cesium. The only elements found that were present in concentrations greater than 1000 ppm were cesium and sodium. All other impurities found in the cesium were either not present in the matrices, found in small insignificant quantities, or could not be distinguished from the constituents of the basic stainless steel.

The residual concentrations of Cs and Na found in the matrices may be due to the accumulation of the metal oxides. Because of the sample preparation techniques used in the spectrographic and metallographic analyses, the alkali metals in the elemental form as well as their oxides, were necessarily exposed to air prior to the analyses. Hence there was no way to distinguish between alkali oxides formed from final exposing of the matrices to air and alkali oxides introduced by the cesium supply or created by internal oxide formations occurring during the vaporization test. The source of these latter oxides is due to either the entrapped oxygen on the interior walls of the stainless steel containment system or infinitesimal air leaks which are inherent in any test rig of this nature.

An estimate of the extent of pore blockage (plugging) is given as follows:

Consider a matrix of length "L" and cross section "A". It is 50 per cent porous, composed of stainless steel with a density of 7.5 gm/cm^3 , and has a cesium and sodium residue analyses the same as the maximum at the liquid inlet section of matrix No. 2 (Table 4-4).

i.e., Na = 0.28 per cent by weight

 Cs = 4.96 per cent by weight

The common oxides of sodium and cesium are respectively Na_2O and Cs_2O . The weight (x) of Na retained in the matrix is, therefore:

$$\frac{x}{0.5 (7.5) AL + x} = 0.0028$$

$$x = 0.009 AL \text{ grams}$$

From the stoichiometric relations between elemental sodium and its monoxide every one gram of sodium can be converted into 1.13 grams of Na_2O .

Assuming that all the sodium is in this oxide form, the weight of Na_2O present is:

$$(1.13) (0.009 AL) = 0.0103 AL$$

$$\text{The density of } \text{Na}_2\text{O} \text{ is } 2.27 \text{ gm/cm}^3 \quad (1)$$

(1) Hodgman, C. D., Weast, R. C., and Wallace, C. W., "Handbook of Chemistry and Physics", Chemical Rubber Publishing Company, Cleveland, Ohio, 35th Edition, 1954.

The volume of Na_2O present is $0.013 \text{ AL} / 2.27 = 0.00498 \text{ AL cm}^3$.

The void volume within the matrix is 0.5 AL cm^3 .

The per cent of the void volume plugged by the sodium oxide is

$$(0.00498 \text{ AL}) / (0.5 \text{ AL}) \times 100\% = 1\%$$

A similar calculation for cesium and its monoxide shows the fraction of the matrix plugged by this substance is 9.5 per cent. The total percentage of plugged volume matrix pore blockage is, therefore, $(1 + 9.5) = 10.5$ per cent, accomplished over a 2100 hour test period. If matrix obstruction from oxide formations were to continue at this same rate, the time for the alkali metal oxides to occupy all the pore volume would be:

$$2100 / 10.5\% = 20,000 \text{ hours} = 27.8 \text{ months}$$

Therefore, it is concluded that neither the sodium build up by itself or this combined with cesium oxide in the matrices, was sufficient to cause any serious short term plugging of the matrix.

From this data, a conservative estimate is made that matrix plugging is not sufficiently serious to hinder the operation of the ion engine for periods up to at least one year. Actually, longer periods of time than this could be expected by merely decreasing the mass flow rate per unit cross section area of the matrix. Also, minute air leaks, although important in laboratory tests, would not be significant in space use. For here the surrounding environment would be a vacuum. The only source of oxygen for the alkali oxide plugs would be the walls of the containing system and the cesium supply. Since this represents a source of only finite quantity it is not probable in space that the pore volume within the matrices would ever be completely plugged solely by alkali oxide build up.

5.4 Metallographic Evaluations

The purpose of the metallographic analyses was to determine matrix grain size, grain growth, a general description of the crystalline structure, and any changes in the structure resulting from cesium contact. In comparing both the macro and the microphotographs in Figures 4-4, 4-5, and 4-6, there is no significant difference between the untested and tested matrices. There did not appear to be a preferential cesium attack at the grain boundaries. Also there appeared

to be no significant difference in the crystalline structure of either the matrices exposed or unexposed to cesium, and there was no evidence of any type of corrosion attack by the alkali metals.

General conclusions made from the macro and microphotographs were:

1. A considerable variation in matrix particle size and configurations was evidenced within any one vaporizer section.
2. The grain size of the particles varied in each of the tested matrices and in the initial control sample.

5.5 Relation of Test Results to Ion Engine Operation

For a feed system to be functional with the operation of an ion engine, it is essential that a continuous non-interrupted propellant flow be supplied to the thruster. One concept for building this feed system utilizes a porous matrix as a vaporizing element. If this element were to become plugged or clogged, the propellant flow would become interrupted and cause engine shutdown.

After 2100 hours of matrix testing with cesium, there appears to be no evidence that this would occur. Matrix deterioration did not happen. Even though some sodium was being concentrated in this matrix, the magnitude of this concentrate was insignificant in comparison with the magnitude of the overall voids of the vaporizers. Also, the x-ray analysis showed no evidence of any impurity build up. From this work alone, it appears that matrix plugging or deterioration is not a significant problem area.

6.0 CONCLUSIONS

The following conclusions concerning the performance of the porous matrix phase separators can be made:

1. During the cesium vaporization process in a matrix at 800°F and 0.5 psia, sodium and probably lithium are concentrated in the matrix while potassium and rubidium are not.
2. After 2100 hours the build up of sodium and possibly cesium oxide, although detectable, is not significant in terms of the overall weight or volume of the matrix. Any plugging resulting from this build up is unlikely.
3. Leaching of stainless steel elements in the matrix does not occur up to at least 2100 hours exposure to cesium vapor at 800°F and 0.5 psia.
4. Type 347 stainless steel matrices are not corroded by cesium vapors at 800°F and 0.5 psia, at exposure times of at least 2100 hours.

In evaluating the suitability of a porous matrix phase separator as a component in an ion engine feed system questions arise other than those examined in this report. Specifically these areas of interest are:

1. The pressure drop across the matrix as a function of cesium boiling rates and operating time.
2. The exact experimental location of the boiling cesium liquid-vapor interface and the stability of this interface over extended test periods.

Because of these questions, further investigations into the long term performance of matrix phase separations is desirable. The course of the suggested studies would be to develop and improve techniques for periodically measuring the exact location of the liquid interface in the vaporizer matrix. Also, means should be established for experimentally measuring the pressure drop across the matrix and measuring the flow rate of cesium passing through each individual matrix. Controls on parameters of this nature should add significantly to the experimental understanding of the boiling and plugging phenomena in porous matrix applications.

DISTRIBUTION LIST FOR FINAL REPORT

CONTRACT NAS3-2518

NASA-Lewis Research Center (1)
Spacecraft Technology Procurement Section
21000 Brookpark Road
Cleveland, Ohio 44135
Attention: John H. DeFord

NASA-Lewis Research Center (1)
Technology Utilization Office
21000 Brookpark Road
Cleveland, Ohio 44135
Attention: John Weber

NASA Headquarters (2)
FOB - 10B
600 Independence Avenue, N.E.
Washington, D. C. 20546
Attention: RNT/James Lazar

NASA-Marshall Space Flight Center (1)
Huntsville, Alabama
Attention: M-RP-DIR/Dr. E. Stuhlinger

Commander (1)
Aeronautical Systems Division
Wright-Patterson Air Force Base, Ohio
Attention: AFAPL (APIE)/Lt. Robert Supp

Jet Propulsion Laboratory (1)
4800 Oak Grove Drive
Pasadena, California
Attention: J. J. Paulson

Electro-Optical Systems, Inc. (1)
125 North Vinedo Avenue
Pasadena, California
Attention: R. C. Speiser

General Electric Company (1)
Flight Propulsion Lab.
Cincinnati, Ohio 45215
Attention: M. L. Bromberg

Ion Physics Corporation (1)
Burlington, Massachusetts
Attention: Dr. S. V. Nablo

Space Technology Laboratories (1)
8433 Fallbrook Avenue
Canoga Park, California
Attention: Dr. D. Langmuir

North American Aviation, Inc. (1)
12214 Lakewood Avenue
Downey, California
Attention: Technical Information Office
Dept. 4096-314

NASA-Lewis Research Center (2)
21000 Brookpark Road
Cleveland, Ohio 44135
Attention: Library

Aerojet-General (1)
Nucleonics Division
San Ramon, California
Attention: Mr. J. S. Luce

Hughes Research Laboratories (1)
Malibu Canyon Road
Malibu, California
Attention: Dr. G. R. Brewer

NASA-Lewis Research Center
Spacecraft Technology Division
21000 Brookpark Road
Cleveland, Ohio 44135
Attention: J. H. Childs (2)
Ross Hieber (18)

United Aircraft Corporation (1)
Research Department
East Hartford, Connecticut
Attention: Dr. R. G. Meyerand, Jr.

TRW Electromechanical Division (1)
Thompson Ramo Wooldridge Inc.
23555 Euclid Avenue
Cleveland, Ohio 44117
Attention: R. T. Craig

NASA-Lewis Research Center
Electromagnetic Propulsion Division
21000 Brookpark Road
Cleveland, Ohio 44135
Attention: W. R. Mickelsen (1)
W. Moeckel (1)

Westinghouse Astronuclear Laboratories (1)
Pittsburgh, Pennsylvania 15234
Attention: Electric Propulsion Lab.
Mr. W. H. Szymanowski

Distribution List (Cont.)

NASA Scientific and Technical Information Facility (6+1)
Box 5700
Bethesda, Maryland 20014
Attention: RQT-2448/NASA Representative

AFWL (1)
WLPC/Capt. C. F. Ellis
Kirtland Air Force Base
New Mexico

NASA-Lewis Research Center (1)
21000 Brookpark Road
Cleveland, Ohio 44135
Attention: Reports Control Office

Aerospace Corporation (1)
P. O. Box 95085
Los Angeles, California 90045
Attention: Library Technical Documents Group